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ADMIRALTY TURBINE OIL O.M.88.

The following paper has been authorized for publication by the Engineer-in-Chief of the Admiralty at whose request and in collaboration with whose department the specification "Turbine Oil O.M.88" issued by the Director of Navy Contracts was devised by the Petroleum Board Lubricating Oil Technical Committee.

The material is summarized from the detailed records of the Committee and its panels.

Introduction.

For many years Admiralty steam turbine lubricating oil was required to meet the Specification "Special Mineral Lubricating Oil, Admiralty Schedule No. 374 (S.M.L.O.)," of which the outstanding features were:

(*a*) the oil should have a specific gravity not greater than 0·880 at 60° F;
(*b*) its sulphur content should be not more than 0·2 per cent;
(*c*) it should not be solvent-refined;
(*d*) it should conform to specified friction-reducing characteristics;
(*e*) it should meet a specified mechanical type of demulsibility test.

These requirements virtually limited the source of supply of Special Mineral Lubricating Oil to such as were derived from the paraffinic-type crude oil of the Pennsylvanian fields.

In peace time, supplies to the Admiralty requirements were readily forthcoming, but with the imminence and advent of war the specification was in some quarters considered unduly and possibly dangerously restrictive. For such reasons the Admiralty, after a series of conferences, expressed themselves as being prepared in emergency to relax the clauses pertaining to specific gravity and solvent refining although, as it turned out, very little use had to be made during the war of such relaxation.

Throughout its long career very little trouble was experienced with the Admiralty Special Mineral Lubricating Oil. A certain amount of corrosion was experienced from time to time, but this was not directly attributable to the oil. Occasional emulsion troubles were generally traced to contamination. In practice, the oil was often discarded before reaching the end of its useful life as a turbine oil and employed for other purposes.

The cycle of marine-turbine lubrication is onerous because of the need for rapid circulation, the exposure of the oil to considerable conducted heat because of the enclosed space, and the severe oxidation conditions arising from particulation of the oil spray when directed to gear wheels and pinions.

These effects are all intensified in naval craft under wartime conditions, and there is therefore a tendency for oils to deteriorate more rapidly. For such reasons it became desirable to give a wider margin of safety to Admiralty turbine oil by adopting the use of oils suitably inhibited against oxidation by means of chemical additives. Extended experience of such oils had already been gained in Merchant-Navy service and in land installations.

Experience of the United States Navy also indicated the need, under some conditions, of being able by means of the oil to control corrosion and rusting, particularly on gear wheels and housings, such as might result from insufficient ventilation under wartime conditions and from the intrusion of

salt water due to damage. U.S. Navy experience with oils suitably treated chemically to have this rust-preventing effect indicated that considerable advantages were to be expected. Corresponding disabilities in the Royal Navy were also experienced, and the Admiralty's wish to have at their disposal oil of the highest performance and such as would be compatible with and at least equivalent to U.S. Navy turbine oil resulted in a decision in late 1944 to have available a revised turbine oil specification for post-war supplies.

The Technical Committee of the Petroleum Board Lubricating Oil Pool was entrusted with the development of such specification, the terms of reference being " to draft a British specification for inhibited turbine oil to provide equivalent performance to the U.S. Navy Department's 2190–T Oil." * For this purpose the Committee co-opted nominated representatives of H.M. Services and Ministries concerned. They also invited further representation from the petroleum industry.

The specification called for has now been completed and accepted by the Admiralty for use as and when required. It has been issued by the Director of Navy Contracts under the title " Turbine Oil O.M.88 " and is reproduced in Appendix I.

The composition of the committee is given in Appendix II.

It will be realized that not only does O.M.88 specification permit the use of chemical additives, but in effect demands them, since some of the important requirements cannot at present be met by plain mineral oils. This applies in particular to anti-oxidants to enable the oil to meet Item 10 (xi) and to rust preventives for Item 10 (viii). Since defoamants may also be required for Item 10 (ix) it is possible that an oil to meet the specification may contain three chemical additives of widely different type.

Description of the Specification.

The reasons for the various changes embodied in the new specification are as follows :—

Specific Gravity.

Limits for specific gravity are omitted from O.M.88 Specification. This is in accordance with modern practice because (*a*) the development of solvent-refining processes makes specific gravity no longer a true guide to the origin and character of an oil and (*b*) in a " performance specification " control of quality is adequately covered by other clauses. In the specification for S.M.L.O. the inclusion of a specific-gravity clause, coupled with the non-admission of solvent-refined oils, virtually limited the source of the oil to Pennsylvanian crudes, refined by older methods. During the war this resulted occasionally in difficulties in obtaining supplies.

Flash Point.

The closed flash point of 330° F in the new specification is somewhat lower than that for S.M.L.O. It corresponds, however, to the open flash point of 350° F in the U.S. Navy Specification for 2190–T oil. Flash point

* Specified in U.S. Bureau of Ships Ad Interim Specification 14-0-15 (INT) (1945).

of mineral lubricating oil bears little relation to fire hazard, and the properties more nearly related to fire hazard are cracking and spontaneous ignition temperatures. The last-named is high for mineral lubricating oils as a whole, but is generally lower for the paraffinic (*e.g.*, S.M.L.O.) types than for the more naphthenic oils which are admissible in the new specification. The spontaneous ignition temperature depends a great deal upon the conditions to which the oil is exposed, and the significance of figures obtained by an arbitrary laboratory test under a given set of conditions would be doubtful.

Viscosity.

The viscosity of the O.M.88 oil is somewhat higher than that of S.M.L.O., the limits for viscosity being based on values suggested by the Admiralty. This increase in viscosity is not considered significant in the lubrication of gears, although for this purpose the trend is in the right direction. The viscosity index may permissibly be lower for O.M.88 oil than for S.M.L.O. Lengthy experience has shown that turbine oils of medium viscosity index can give excellent performance and response to additives so that the wider range in that respect now admitted ensures increased source of supply.

Sulphur Content.

A limit for sulphur content has not been included in the new specification. Some of the chemical additives currently used for imparting resistance to oxidation may contain sulphur chemically combined in the molecule as do certain natural anti-oxidants in petroleum. Limitation of sulphur content might, therefore, limit the presence of these desirable constituents.

The oil is, however, required to be free from corrosive sulphur compounds.

Corrosion.

Besides being free from corrosive sulphur compounds the O.M.88 oil is designed to possess anti-rust properties and to limit corrosion of steel by salt water. A test has, therefore, been included to determine the extent of protection given by the oil against simulated sea water, and the limits specified are such as are likely only to be met by the use of chemical additives.

The additives effective in imparting resistance to rusting are generally polar in nature and probably act by virtue of adsorption at the metal surface, thereby preventing access of water, and not merely by modificaton of the interfacial tension. For this reason a used oil may not apparently possess full anti-corrosion properties, since the additive may have already concentrated at the metallic surfaces involved.

Ash Content.

No limit has been specified for ash, but its determination may be conducted for routine inspection purposes. It is considered that limitation of ash content might tend to hamper the development of useful chemical additives.

Demulsification.

The old Admiralty (Phillips) test has been replaced by the Institute of Petroleum method for determining "Demulsification Number." The former involves mechanical mixing and circulation, but though it gives fair repeatability in the hands of experienced operators it is unduly sensitive and can be upset by the slightest contamination, so that its reproducibility is poor. Moreover, since a demulsification test is required on the oil after oxidation the I.P. method is preferable, the Admiralty test not having been worked out for application to used oils. Also, the Admiralty test requires a large quantity of oil, and this would impose particular difficulty in carrying out the oxidation test.

The I.P. test is the standard procedure in use throughout the oil industry in the U.K. and U.S.A., and is considered particularly suitable for turbine oils. It is rapid and accurate and requires a comparatively small amount of oil. It is also possible to test used turbine oils by this means, and useful data have been accumulated in this respect over many years.

The requirements set for new and oxidized oils are derived from results obtained on samples of U.S. Navy 2190–T oils before and after oxidation, and include satisfactory operating margins. It is to be noted, however, that the limits imposed for new oils are higher than those for corresponding plain mineral oils, since the anti-rust additives tend to slow up the separation of oil from water.

Friction Reduction.

A friction test has not been included in the new specification as it is considered that petroleum oils possess adequate friction-reducing qualities for turbine lubrication and differ little amongst themselves in this respect. It is to be noted that the anti-rust additives commonly used in new-type turbine oils tend to reduce the coefficient of static friction of mineral oils.

Foaming Behaviour.

In order to control the tendency of additive-treated oils to "foam" in service, a new test has been introduced in the O.M.88 specification. This test is virtually of the "go" or "no-go" type. In the absence of better procedure, the method currently used for 2190–T oil has been adopted in a slightly abbreviated form. The Institute of Petroleum is investigating the possibility of developing a more satisfactory test.

Resistance to Oxidation.

A method to differentiate in respect of oxidation stability between oils of the 2190–T type and plain mineral oils was clearly necessary, and a recommendation for an interim test pending further investigation has been made as the result of extensive examination in a number of laboratories, using a variety of methods.

The test chosen is a modification of the Michie test for sludging value originally developed for electrical transformer oils. This modified test has now been standardized by the Institute of Petroleum.* With a view to

* I.P. Method 114/47 (P).

expediting the obtaining of results, work is being continued on other tests to reduce the time taken.

The criterion of resistance to oxidation is the development of organic acidity as expressed by the neutralization value. A limit of 0·2 has been fixed as this has been found by experiment to be a satisfactory line of demarcation between 2190–T-type and uninhibited oils. In the oxidation of inhibited oils there is an induction period during which the development of acidity is slow, but after which the development is more rapid. The end of this induction period is commonly used as the criterion for changing the lubricating oil in a system. It is considered that the limit for acidity suggested is within the induction period of 2190–T-type oils whereas plain mineral oils develop acidity markedly above 0·2 within the same space of time.

In addition to acidity, the demulsification value after oxidation is measured, and a maximum figure has been chosen which again appears to distinguish between the two types of oil.

It is to be appreciated that these criteria refer only to the oil after it has been submitted to artificial oxidation and are not necessarily related to its behaviour in service. The Admiralty will seek to correlate these data with those obtained in actual performance, and such information, when obtained, will be made available to the oil industry.

General.

In addition to the physical and chemical properties referred to in the foregoing, general clauses have been introduced into the O.M.88 specification to cover such factors as description of equipment in which the oil will be used in service. The Admiralty will arrange where necessary for details of such equipment to be made available to suppliers.

General clauses have been introduced into the specification to ensure uniform distribution of the additives throughout the oil. Behaviour in this respect is covered at temperatures from 10° F above the maximum pour point specified up to 250° F. It is recognized that the action of higher temperatures on the solubility and response of the additives will have to be the subject of further investigation. When sufficient data are acquired it is hoped to be possible to devise suitable tests for inclusion in para. 10 of the specification.

The possible action of centrifugal purification in affecting uniform additive distribution has also been considered. This, too, requires further investigation, which should be directed especially towards study of the effect of higher temperatures in the absence of water-washing, since it is not practice in the Royal Navy to wash the oil before or during centrifuging.

The oil industry has seen no objection to the disclosure to the Admiralty of the amount and nature of the additives used and the origin and method of refining of the oil. The Admiralty has stated that such information will be regarded as confidential.

Although it is the intention of the Admiralty to approve separately the additives which may have been used it is stated that final approval will be given to the finished oil, *i.e.*, mineral oil plus additive or additives. Since individual suppliers cannot be expected to be familiar with all alternative additives supplied, decision as to compatibility will rest with the Admiralty.

Selection of Test Methods.

Oxidation Test.

A large number of test methods in general use or reported in literature was considered for suitability in respect of the following requirements :—

(*a*) that the test period be of short duration;
(*b*) that the test make use of standard apparatus;
(*c*) that the results be well reproducible;
(*d*) that the test give a clear distinction between oils approved as 2190-T oils and high grade mineral turbine oils.

From the more promising methods, variations of four main types were examined, comprising air blowing, oxygen absorption, bomb, and beaker tests. The oils examined included high-grade mineral oils, including S.M.L.O., and approved 2190–T oils.

After exploration in various co-operating laboratories of a number of tests of these four types, a modification of the Michie sludge test in respect of time and temperature was considered most adequately to satisfy the requirements. A great deal of experimental work on this particular test method was carried out before the details were finally settled, and various methods of accelerating the reaction were tried out with interesting results; *e.g.*, in one series of experiments a soluble catalyst (ferric stearate) was used but gave misleading results, some 2190–T oils appearing relatively bad, whilst some mineral oils appeared outstandingly good. The reason for this behaviour in the case of inhibited oils was thought to be due to interaction in some cases with the anti-oxidant or anti-rust additive. Moreover, the soluble catalyst interfered with the subsequent examination of the oxidized oil for demulsibility.

The temperature of the test precluded the presence of water if this were added as a standard condition of the test, but in order to provide for the possibility that water would affect the additives a preliminary water-washing was included. In the case of the 2190–T oils tested, this had no significant effect on the oxidation results with any of the reference oils. The washing procedure was to agitate the oil and water by a mechanical stirrer for a period of thirty minutes at 90° C.

Table I gives the results in various laboratories on two mineral oils and five 2190–T oils by the modified Michie test finally adopted, both before and after treatment with water.

It will be noted that apart from two rather odd results on one of the oils, all 2190–T oils gave neutralization numbers of the order of 0·1 or lower, whilst the mineral oils gave figures of 0·3 and upwards. As the asphaltene formation was in all cases negligible, it was not considered necessary to legislate for asphaltene content.

Demulsification Test after Oxidation.

The Admiralty expressed a wish to incorporate a test for demulsification on the oxidized oils. The desiderata in selecting such a test were as follows :

(*a*) simplicity of operation;
(*b*) standard apparatus available in the U.K.;
(*c*) relatively small quantity of oil required.

TABLE I.

Oxidation Tests by the Method Finally Adopted (I.P.—114/47 (P)).

Ref. no.	Type of oil.	Laboratory.	Oxidation tests on oil untreated with water. (Tests after oxidation.) Acidity (I.P. 1A/44).	Asphaltenes (I.P. 6/45).	Demulsification number (I.P. 19/42).	Oxidation tests on oil after treatment with water. (Tests after oxidation.) Acidity (I.P. 1A/44).	Asphaltenes (I.P. 6/45).	Demulsification number (I.P. 19/42).
1	S.M.L.O.	A	0·30	Trace	1200 +	—	—	—
		B	0·52	Trace	1200 +	—	—	—
		C	0·45	0·04	1200 +	—	—	—
		D	0·48	Trace	1200 +	—	—	—
		E	0·45	Trace	1200 +	—	—	—
3	2190-T oil	A	0·05	Nil	600	0·08	—	570
		B	0·03	Nil	435	0·03	0·05 †	420
		C	0·05	0·02	450	—	—	—
		D	0·06	Trace	480	—	—	—
		E	0·06	Trace	510	0·06	Trace	510
4a*	2190-T oil	A	0·10	Nil	400	0·10	—	520
		B	0·20	Nil	420	0·11	Nil	510
		C	0·10	Nil	330	—	—	—
		D	0·14	Nil	420	—	—	—
		E	0·17	Nil	405	0·22	Nil	480
4b*	2190-T oil	A	0·09	Nil	480	—	—	—
		B	0·13	Nil	390	0·08	Nil	410
		C	0·10	Nil	340	—	—	—
		D	0·08	Nil	510	—	—	—
		E	0·14	Nil	380	0·06	Nil	420
5	Uninhibited solvent-extracted distillate (high V.I)	A	0·30	Nil	1200 +	—	—	—
		B	0·40	Nil	1200 +	—	—	—
		C	0·35	Nil	1200 +	—	—	—
		D	0·25	Nil	1200	—	—	—
		E	0·45	Nil	1200 +	—	—	—
6	2190-T oil	A	0·05	Nil	475	0·08	—	490
		B	0·03	Nil	555	0·03	Nil	900
		D	0·05	Nil	420	—	—	—
		E	0·06	Nil	405	0·06	Nil	660
7	2190-T oil	A	0·08	Nil	585	0·05	—	530
		B	0·08	Nil	570	0·03	Nil	390
		C	0·10	Nil	1200	—	—	—
		D	0·08	Nil	660	—	—	—
		E	0·11	Nil	315	0·11	Nil	420

* Oils 4a and 4b represent two shipments of the same oil.
† Precipitation of inhibitor.

The procedure adopted distinguishes between 2190–T oils and mineral oils, but it is uncertain whether much is gained otherwise by introducing this requirement in a specification, as deterioration in demulsibility in the cases examined seems to follow the same course as other properties of the oil during oxidation. Moreover, the reproducibility of the test is not particularly good.

Results on unoxidized oils are in general higher for 2190–T oils than for mineral oils, as is shown in Table II.

Table I includes the results for demulsification number on the oxidized oils.

Salt-water-corrosion Test.

No well-developed corrosion test had been worked out in the U.K., but the test laid down in U.S.N. Specification 14-0-15 is an easy one to carry out

TABLE II.

Demulsification Numbers of Fresh Oils—I.P. 19/42.

Ref. no.	Type of oil.	Laboratory.				
		A.	B.	C.	D.	E.
1	S.M.L.O.	160	215	180	150	150
2	2190-T oil	230	245	260	210	275
3	2190-T oil	135	135	190	120	150
4	2190-T oil	170	190	210	180	195
4*a**	2190-T oil	210	210	200	210	210
4*b**	2190-T oil	230	210	240	210	225
5	Uninhibited solvent-extracted distillate (high V.I.)	130	120	120	150	105
6	2190-T oil	150	180	180	180	165
7	2190-T oil	225	255	230	180	180

* Oils 4*a* and 4*b* represent two shipments of the same oil.

and has been reported from the U.S. as reasonably reproducible. Accordingly work was carried out on the reference oils using simulated sea-water of specified composition instead of distilled water. This was found to discriminate quite clearly between the two classes of oil, and the test method was adopted as it stood, with the modification of the details in terms of easily available British materials, *i.e.*, 0·18 per cent carbon bright mild-steel rod cleaned by rubbing first with No. 0 and then with No. 00 emery paper until free from any visible scratches, and finally rubbed down with a pad of cotton wool before immersion in the oil. The test is preceded by water-washing as in the case of the oxidation test in order to guard against the use of rust preventives affected by water.

Table III gives results obtained by two laboratories on S.M.L.O. and several 2190–T oils after they had been treated with water.

Foaming Test.

In the absence of any recognized foaming test in the U.K. the 2190–T reference oils were tested according to the U.S. Co-ordinating Research Council (CRC) Designation L-12-445 Test Procedure for Determining Foaming Characteristics of Lubricating Oils. It was found, however, that most of the oils did not pass the full requirements of this test which is laid down as part of U.S. Navy (Bureau of Ships) Ad Interim Specification 14-0-15 (INT) for Oil Lubricating Turbine, Non-Corrosive. It should be noted, however, that this is possibly due to the fact that some 2190–T oils may not have been treated with foam inhibitor as they may have been supplied to an earlier version of Specification 14-0-15 (INT) which did not require this.

Reproducibility was very bad, variations of as high as 800 per cent having been found by different laboratories.

The results of tests on two straight mineral oils and seven 2190–T oils are listed in Table IV.

TABLE III.

Salt Water Corrosion Tests Before and After Treatment with Water (14-0-15 (*INT*) *Modification of A.S.T.M. D665–44T*).

Ref. no.	Type of oil.	Untreated oil.	Treated oil.	
		Laboratory C.	Laboratory C.	Laboratory E.
1	S.M.L.O.	Severe rusting	Severe rusting	—
2	2190-T oil	Satisfactory	Satisfactory	Satisfactory
3	2190-T oil	Slight rust above oil surface	Slight rust above oil surface	Satisfactory
4	2190-T oil	Satisfactory	Light general rusting	Slight pitting
4*a* *	2190-T oil	Satisfactory	—	—
4*b* *	2190-T oil	Pin-points of rust	—	Satisfactory
6	2190-T oil	Satisfactory	—	Satisfactory
7	2190-T oil	Satisfactory	—	Satisfactory

* Oils 4*a* and 4*b* represent two shipments of the same oil.

TABLE IV.

Foaming Tests—CRC Designation L-12-445.

Ref. no.	Type of oil.	Laboratory.	Sequence I.		Sequence II.		Sequence III.	
			5-min. foaming.	10-min. foam collapse.	5-min. foaming.	10-min. foam collapse.	5-min. foaming.	10-min. foam collapse.
1	S.M.L.O.	A	580	*350*	60	Nil	550	80
		C	740	180	80	Nil	760	*340*
		D	136	Nil	35	Nil	130	Nil
2	2190-T oil	C	670	200	100	Nil	610	190
3	2190-T oil	A	350	Nil	40	Nil	430	20
		C	460	Nil	60	Nil	430	Nil
		D	6	Nil	5	Nil	5	Nil
4	2190-T oil	A	630	*550*	500	Nil	610	*550*
		C	580	*530*	550	Nil	750	*720*
		D	95	70	95	Nil	144	120
4*a**	2190-T oil	A	600	*490*	—	—	—	—
		C	680	*610*	160	Nil	770	730
4*b**	2190-T oil	A	310	150	—	—	—	—
		C	(590)	(*420*)	(770)	(*430*)	(650)	(*610*)
			(620)	(*430*)	(850)	(*500*)	(750)	(*700*)
5	Uninhibited solvent-extracted distillate (high V.I)	A	660	*360*	—	—	—	—
		C	660	280	70	Nil	730	*410*
6	2190-T oil	A	400	140	—	—	—	—
		C	660	280	70	Nil	730	*450*
7	2190-T oil	A	Nil	Nil	—	—	—	—
		C	Nil	Nil	50	Nil	Nil	Nil
	Specification		—	300 max.	—	25 max.	—	300 max.

The readings in italics are outside specification.

* Oils 4*a* and 4*b* represent two shipments of the same oil.

It is possible that those 2190-T oils failing to pass the specification had not been treated with foam inhibitor, as they may have been supplied to an earlier version of specification 14-0-15 (INT) which did not require this.

APPENDIX I.

ADMIRALTY SPECIFICATION FOR TURBINE OIL O.M.88.

1. Turbine lubricating oil is intended to be used in the following equipment in service :—

(*a*) main turbines and gears;
(*b*) auxiliary turbine installations;
(*c*) hydraulic systems and general mechanical equipment.

2. Turbine lubricating oil shall consist of refined petroleum hydrocarbons with or without additive agents.

3. Turbine lubricating oil shall be free from water, mineral acidity, natural fats or fatty oils, suspended matter or sediment.

4. Additive agents where used shall remain uniformly distributed throughout the oil at all temperatures between 20° F and 250° F. The oil shall not be affected by centrifugal purification in the absence of water washing.

5. If the oil is cooled below its pour point it shall regain its homogeneity on standing at a temperature of 30° F.

6. The geographical origin, quantity and chemical nature of the additives when used shall be disclosed to the Admiralty inspecting authority, together with any method used of determining the quantity of additive in the oil. This information will be regarded as confidential.

7. Additives will be subject to prior approval by the Admiralty.

8. The general nature of the oil shall be stated, together with its geographical origin and the type of process or processes by which it has been refined. This information will be regarded as confidential.

9. The oil shall qualify in respect of compatibility both when mixed with other unused oils supplied to this specification and also when added to such oils which are already in use.

10. Turbine lubricating oil shall conform to the following physical and chemical characteristics :—

(i) Closed flash point (I.P. 34/46; A.S.T.M. D93–42)	Minimum 330° F
(ii) Viscosity at 100° F (I.P. 71/45 (T); A.S.T.M. D445–42T)	82–92 cs
(iii) Viscosity Index (I.P. 73/42; A.S.T.M. D567–41)	Minimum 50
(iv) Pour Point (I.P. 15/42; A.S.T.M. D97–39)	Maximum 20° F
(v) Acidity (I.P. 1/46 Procedure A)	Maximum 0·1 mg KOH/g
(vi) Ash content	Ash content may be determined for control purposes
(vii) Sulphur (Corrosive) (I.P. 64/46 Procedure B)	Must pass
(viii) Salt-water-corrosion test (Appendix A)	Must pass
(ix) Foaming test (Appendix B)	Must pass
(x) Demulsification Number (I.P. 19/42)	Maximum 300
(xi) Resistance to Oxidation (Appendix C). After oxidation the oil shall comply with the following :—	
(*a*) Acidity (I.P. 1/46 Procedure A)	Maximum 0·2 mg KOH/g
(*b*) Demulsification number (I.P. 19/42)	Maximum 1200

Appendix A.

Method A.S.T.M. 665–44T, modified as follows :—

Salt water of the following composition :

Magnesium chloride ($MgCl_2 6H_2O$)	11·0 grams per litre
Anhydrous calcium chloride ($CaCl_2$)	1·2 „ „ „
Anhydrous sodium sulphate (Na_2SO_4)	4·0 „ „ „
Sodium chloride (NaCl)	25·0 „ „ „

The steel specimen used in the test shall be of 0·18 per cent carbon bright mild steel. The rod shall be cleaned by rubbing first with No. 0 and then with No. 00 emery paper until free from any visible scratches. It shall finally be rubbed down with a pad of clean cotton wool and immersed in the oil.

Prior to this test 250 g of the oil shall be stirred with 50 g of distilled water for 30 minutes at 1000 r.p.m. at 90° C using the beaker and stirrer specified in A.S.T.M. Rusting Test D665–44T. After 30 minutes stirring the mixture shall be transferred to a separating funnel and left to separate when the water layer shall be drawn off.

Appendix B.

CRC Designation L-12-445 (Sequences 2 and 3 only).

Sequence 2: No maximum limit immediately following the 5-minutes bubbling period, but a maximum limit of 25 ml after 10 minutes of foam collapse.

Sequence 3: No maximum limit immediately following the 5-minutes bubbling period, but a maximum limit of 300 ml after 10 minutes of foam collapse.

Appendix C.

Method I.P. 56/46 modified in respect of temperature −110° C, and time −90 hr, but omitting all procedure subsequent to para. 4 (*h*). Prior to the test the oil shall be treated as in para. 3, Appendix A.*

NOTE: Only oil treated as in paragraph 3, Appendix A, separated from the water is to be used to carry out the tests called for in Section viii and xi of paragraph 10 of the specification.

APPENDIX II.

PETROLEUM BOARD LUBRICATING OIL TECHNICAL COMMITTEE.

COMPOSITION OF COMMITTEE.

Lt.-Col. S. J. M. Auld (*Chairman*)
L. O. Maskell.
R. D. Streeton (or E. H. Wild).
R. S. Teale.
E. A. Evans (or J. C. Cragg).
J. M. Wilson.
J. L. Taylor (*Secretary*).

Admiralty and War Office.

Engr.-Cdr. L. J. Le Mesurier, E-in-C's Dept., Admiralty.
Engr.-Capt. H. Moy, E-in-C's Dept., Admiralty (In succession).
Cdr. (E) R. T. Sandars, E-in-C's Dept., Admiralty (In succession).
Lt.-Cdr. (E) E. Tyrrell, E-in-C's Dept., Admiralty (In succession).
Dr D. Wyllie, Admiralty Chemical Department.
Dr D. Clayton, M.O.S. (CRD6) and Admiralty.
Lt.-Col. H. F. Jones, War Office, S.T. 2(e).
Major W. G. Kyle, War Office, S.T.2(e) (In succession).

Co-opted.

P. W. L. Gossling
V. Biske.
G. H. Harries (or N. A. Clegg).
Dr N. L. Anfilogoff.
Dr H. Weiser
W. Twizell.

Oil Tests Panel.

R. S. Teale (*Chairman*).
J. C. Cragg.
P. W. L. Gossling.
L. O. Maskell.
R. D. Streeton.
Dr D. Wyllie.
Dr N. L. Anfilogoff. } Part time.
Dr C. B. Davies. } Part time.
T. M. Taylor. } Part time.
E. H. Wild. } Part time.

* This method has since been published as I.P.—114/47 (P).

SOME SUBSTITUTION PRODUCTS OF THE LIQUID ISOMER OF THIOPHTHEN—THIOPHENO-2′ : 3′ (OR 4′ : 3′) : 2 : 3-THIOPHEN.

By Frederick Challenger (Fellow), Peter H. Clapham, and Robert Emmott.

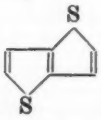
Summary.

Several new mono-substitution derivatives of the liquid isomer of thiophthen have been prepared, including iodothiophthen, methyl thiophthienyl ketone, the corresponding carboxylic acid and its methyl and *p*-nitrobenzyl esters. Friedel–Crafts and Grignard reactions, and also iodine have been shown to cause substitution in the same position in the ring. This, as in the case of the solid isomer of thiophthen, is contrary to the behaviour observed with thionaphthen. The orientation of these derivatives is as yet unknown.

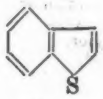
The liquid isomer prepared from acetylene and boiling sulphur has been proved to be identical with thiophthen obtained from citric acid and "phosphorus trisulphide" by the comparison of several analogous derivatives.

Challenger and Harrison [1] showed that the thiophthens $C_6H_4S_2$ obtained by earlier workers [2,3] by the interaction of acetylene and boiling sulphur consisted of two, and possibly three, isomers. From the fraction of b.p. 221° to 222° which had been purified through the non-separable mixed picrate of m.p. 139° to 141° and regenerated by sodium carbonate a solid thiophthen (T) was isolated by repeated freezing. This was shown by X-Ray examination and by its zero dipole moment to consist of thiopheno-2′ : 3′ : 3 : 2–thiophen, m.p. 56°, of the structure (I). The liquid portion of the oil from the picrate, after removal of a small quantity of thionaphthen (II) and a product (X) which resembled the thiophthens but was readily polymerized and blackened by aqueous mercuric chloride, consisted of an isomeric thiophthen (S) of m.p. 6·5°. (Details of the removal of these two last-named products are given in the experimental part.)

Challenger and Harrison [1] showed that most, if not all, of any remaining solid isomer (T) was removed from the liquid thiophthen (S) by fractional crystallization of the styphnate. This second isomer is either thiopheno-2′ : 3′ : 2 : 3–thiophen (III) or the 4′ : 3′ : 2 : 3–derivative (IV) or possibly, though this is regarded as improbable, a non-separable mixture of these two compounds. It is possible that the very reactive substance (X) [4] may be the isomer (IV), but this has not been proved. An attempt by Dr S. A. Miller in these laboratories to prepare the analogous compound (V) from *o*-xylylene sulphide (VI) by dehydrogenation with sulphur failed. No product which blackened with aqueous mercuric chloride could be isolated.



(I)



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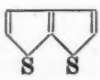
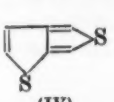
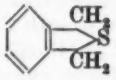


(III)

A liquid thiophthen (C) is also obtained [5,6] by the distillation of citric acid or aconitic acid with " phosphorus trisulphide." From its method of


(IV)

(V)


(VI)

formation this cannot contain the solid isomer (T),[1] but may have either of the structures (III) or (IV) or conceivably be a mixture of both. Compounds of such similar structure would probably not be separable by crystallization of the picrates or styphnates as was shown for the mixture of (T) and (S), see above. Furthermore, a mixture of thionaphthen and " thiophthen " picrates cannot be separated by such means,[7] and benzene and thiophen are known to form mixed crystals. There is, however, no evidence against the homogeneity of (S) and (C), and this will be assumed throughout this discussion. It remained, nevertheless, to determine whether or no they are identical. This point was considered at length by Challenger and Harrison, who found that corresponding derivatives frequently showed slight differences in m.p. although the depression of m.p. on mixing was negligible.

It seemed possible, however,[4] in spite of repeated crystallization of the picrate and styphnate that liquid thiophthen (S) might still have contained traces of the solid isomer (T). This was suggested by the formation of a greenish-blue colour during the polymerization of even the most carefully purified samples of (S) by means of hydrogen bromide in glacial acetic acid. This colour was never observed in the course of the polymerization of citric acid thiophthen (C), which, as already stated, must be free from the solid isomer (T). The bluish-green colour is, however, readily produced on addition of a trace of (T) to (C). It was therefore decided to submit the purest liquid thiophthen (S) from the sulphur–acetylene reaction, from which thionaphthen had already been removed, to a still more rigorous purification through the styphnate. (The purity of this specimen approximated to that of the product (S_4) of Challenger and Harrison.[1])

The styphnate was repeatedly crystallized from alcohol, the m.p. being unchanged and constant at 130° to 131°, until a portion, on decomposition with sodium hydroxide, yielded a thiophthen (S_5) which gave no trace of blue or green colour on polymerization as before. The specimen (S_5) was therefore regarded as being free from the solid isomer. In accordance with this conclusion it was found that the picrate, styphnate, the methyl thiophthienyl ketone, $C_6H_3S_2{\cdot}COCH_3$, the corresponding thiophthencarboxylic acid, $C_6H_3S_2{\cdot}COOH$, and its methyl ester were indistinguishable by m.p. and mixed m.p. from the corresponding derivatives prepared from liquid " citric acid thiophthen " (C_1) purified as described on p. 929. The ketones were oxidized to the acids by means of iodine and sodium hydroxide.

These observations, it is concluded, remove any doubt as to the identity of " sulphur acetylene thiophthen " and " citric acid thiophthen." Further evidence confirmed this conclusion.

It may be added that the slightly lower dipole moment observed [1] for liquid thiophthen obtained from the sulphur–acetylene reaction as com-

pared with that of " citric acid " thiophthen is explained if some of the solid isomer, which has a zero dipole moment, was present.

The two specimens of the carboxylic acids were also identical in m.p. and mixed m.p. with the thiophthencarboxylic acids obtained from the liquid (S_5) and (C_1) thiophthens by the action of magnesium ethyl bromide followed by treatment with carbon dioxide.

It therefore follows that the introduction of the CH_3CO– group by the Friedel–Crafts reaction and of the –MgBr group occurs in the same position in the molecule of liquid thiophthen. The introduction of iodine by the action of iodine and mercuric oxide on liquid thiophthen (S_5) also occurs in the same position. The resulting mono–iodo- derivative gives a Grignard reagent which with acetonitrile yields a methyl thiophthienyl ketone identical in m.p. and mixed m.p. with those obtained as above by the Friedel–Crafts reaction. The formation of the dinitrophenylhydrazone confirmed this observation. Similar relations were established for solid thiophthen [8] though in neither case was the orientation of the groups established. These authors drew attention to the fact that in these respects solid thiophthen behaves differently from thionaphthen (II). Substitution in this compound by electrophilic reagents occurs in the β- or 2-position in the thiophen nucleus. Two exceptions are furnished by sodium and magnesium ethyl bromide, treatment with these reagents followed by the action of carbon dioxide, giving the α- or 1-carboxylic acid. Details and references are given by Challenger and Miller.[9]

In the case of magnesium ethyl bromide, substitution may be indirect and preceded by co-ordination of C_2H_5MgBr on the sulphur atom, followed by elimination of ethane. A similar suggestion was put forward by these authors [10] to explain the exclusive *o*-substitution observed when anisole and phenetole are heated with magnesium ethyl or *n*-propyl bromides.

For electrophilic reagents, therefore, substitution in the two isomeric thiophthens occurs in the same (as yet unoriented) position, whereas in thionaphthen sodium and magnesium alkyl bromides cause substitution in position 1, other electrophilic groups entering at 2. One reservation may, perhaps, be made. In the reaction between the isomeric thiophthens and the Grignard reagent some formation of a dicarboxylic acid occurs. The removal of this compound causes some loss of the monocarboxylic acid. It is therefore conceivable that a second monocarboxylic acid might have escaped notice owing to the small quantities of the thiophthens involved. The work will therefore be repeated using quantities large enough to permit of careful fractional crystallization. The mono-iodo- derivative of liquid thiophthen (S_4), m.p. 55·5°, is a stable compound, see p. 928 obtained by the action of iodine in benzene solution in presence of mercuric oxide, Bruce, Challenger, Gibson, and Allenby [4] found that the analogous reaction with solid thiophthen gave a di-iodo- derivative, but chiefly a brown polymeric substance. If liquid thiophthen (S_4) containing traces of the solid isomer is used the iodo-derivative easily polymerizes after about 12 hr giving an almost black product.

Table I summarizes the evidence on which the identity of liquid thiophthen from sulphur and acetylene with that from citric acid has been established. It will also be seen from the recorded m.p. that substitution by I, $-COCH_3$, and –MgBr occurs in the same position in liquid thiophthen.

TABLE I.

Comparison of the Melting Points of Derivatives of Liquid Thiophthen Prepared (a) *from Sulphur and Acetylene,* (b) *by Rigorous Purification of the Product from* (a) *through the Styphnate, and* (c) *from Citric Acid.*

Derivative.	Liquid thiophthen S/C_4H_4 (trace S thiophthen), S_4.	Pure S/C_4H_4 thiophthen, S_5. (Liquid.)	Pure citric acid thiophthen, C_1. (Liquid.)
1. Picrate	137–138°	137·5°	137·5°
2. Styphnate	130–131°	130–131°	130–131°
3. Methyl thiophthienyl ketone	114–115°	117°	117°
4. Methyl thiophthienyl ketone 2 : 4-dinitrophenylhydrazone	—	267–268°	—
5. Thiophthencarboxylic acid (KL) (from oxidation of the ketone)	244° (d)	245° (slight d)	245° (slight d)
6. Methyl thiophthencarboxylate (KL)	104–105°	106·5°	106·5°
7. Thiophthencarboxylic acid (GL) (prepared by Grignard reaction)	243° (d)	245° (slight d)	245° (slight d)
8. Methyl thiophthencarboxylate (GL)	104–105°	106·5°	—
9. Iodothiophthen	—	55·5°	—
10. Methyl thiophthienyl ketone 2 : 4-dinitrophenylhydrazone prepared from iodothiophthen	—	265–266° Mixed m.p. with (4) was 267°	—

It will be seen that the difference in m.p. caused by the presence of traces of derivatives of solid thiophthen in derivatives of the liquid isomer is very slight.

EXPERIMENTAL.

The Monocarboxylic Acid of Liquid Thiophthen 2′ : 3′ (or 4′ : 3′)-thiopheno-2 : 3-thiophen. Preparation of the Acid (KL) by oxidation of the Methyl Ketone.

The thiophthen was prepared from acetylene and boiling sulphur, purified by fractionation and formation of the mixed picrate of liquid and solid thiophthens [1] and freed from all but traces of the solid isomer by freezing and formation of the styphnate m.p. 127° to 129°, see pp. 922 and 923. Thiophthen was regenerated from this with sodium carbonate, and twice treated with saturated aqueous mercuric chloride to remove the impurity which gives a black partially mercurated polymeric substance [4] under these conditions (see p. 922). The resulting product boiled at 106° to 107° at 19 mm and was no longer blackened by saturated aqueous mercuric chloride. Analysis showed a sulphur content 3 per cent too low, due to the presence of thionaphthen. This was removed by alcoholic mercuric chloride, the thiophthen being mercurated, whereas thionaphthen is inert under these conditions.[1] The separation was accelerated by adding yellow mercuric oxide whereby liberated hydrogen chloride was removed. Thiophthen (83 g) and mercuric oxide (68 g; 0·5 mol) were added to 1690 cc of a saturated solution of mercuric chloride in 80 per cent alcohol (2 mol $HgCl_2$). After 48 hr at room temperature all the oxide had disappeared. The mercurichloride (200 g) was too insoluble for recrystallization; decomposition with dilute hydrochloric acid gave thiophthen (S_4) (52 g;

b.p. 102° at 16 mm; Challenger and Harrison give b.p. 101° to 102° at 17 mm). (Found: C, 51·6; H, 3·1; S, 45·6. Calc. for $C_6H_4S_2$: C, 51·4; H, 2·9; S, 45·7 per cent.) The mother liquors from the first mercuration yielded 7·3 g of pure thionaphthen, m.p. and mixed m.p. 31°. 5 g of the thiophthen (S_4 see p. 923) were again mercurated as before giving 3·5 g of regenerated thiophthen, b.p. 104° at 18 mm. The picrate melted at 137° to 138° as stated by Challenger and Harrison, whereas that prepared from " citric acid thiophthen " melted at 136° to 137°.

Thiophthen S_4 was converted to the methyl ketone, m.p. 108°, which crystallized badly from alcohol owing to the presence of small quantities of high-melting polymeric product. (Traces of such products are always produced during the Friedel–Crafts reaction with thiophthen.) Distillation in steam gave almost pure ketone, m.p. 113°, which was recrystallized from alcohol. The final m.p. 114° to 115° was not affected by two further crystallizations. Challenger and Harrison's [1] purest specimen (S/C_2H_2 method) melted at 113°, and their methyl ketone prepared from " citric acid thiophthen " had m.p. 115° to 116° all taken simultaneously.

The methyl thiophthienyl ketone was oxidized to the carboxylic acid (KL) by the method of Challenger and Gibson.[8] Quantities: ketone 0·5 g., N NaOH 25 cc; iodine solution (13·82 g iodine, 25 g potassium iodide in 250 cc water) 42 cc. Time of addition 2 hr; left overnight. The crude product crystallized from chloroform in fine white needles (0·1 g; m.p. 242°). A second experiment gave 1·9 g acid from 2·2 g ketone. Successive recrystallizations from water, dilute alcohol, chloroform, and twice from water gave products of m.p. 244° to 246°, 244° to 246°, 246° to 247°, 247°, and 247°. (Found: C, 45·9; H, 2·3; S, 34·6; $C_7H_4O_2S_2$ requires C, 45·7; H, 2·2; S, 34·8 per cent.)

The p-*nitrobenzyl ester* was prepared by the method described by Challenger and Gibson,[8] for the isomeric derivative of solid thiophthen, and crystallized from alcohol in large white needles, m.p. 142° to 143°. Three further crystallizations did not alter this figure. (Found: C, 53·0; H, 2·85; N, 4·6; S, 20·6, 20·2. $C_{14}H_9O_4NS_2$ requires C, 52·7; H, 2·8; N, 4·4; S, 20·1 per cent.) The *methyl ester* was prepared with diazo-methane. When crystallized from alcohol the constant m.p. was 104° to 105°. (Found: C, 48·5; H, 3·2; S, 32·5; $C_8H_6O_2S_2$ requires C, 48·5; H, 3·0; S, 32·3 per cent.)

Preparation of Thiophthencarboxylic acid (GL) by means of the Grignard Reagent. The interaction of liquid thiophthen (S_4) with magnesium ethyl bromide in dimethylaniline followed by treatment with carbon dioxide (for 4 hr instead of overnight) was carried out as described [8] for solid thiophthen. The acid (1·5 g) was crystallized first from chloroform and had m.p. 242° which was raised to 245° to 246° (constant) on two crystallizations from water. (Found: C, 45·8; H, 2·4; S, 35·5, 35·6; $C_7H_4O_2S_2$ requires C, 45·7; H, 2·2; S, 34·8 per cent.) In admixture with the acid (KL) from the ketone, m.p. 247°, the m.p. was 245° to 246°.

About 0·2 g of a brown acid insoluble in chloroform and decomposing about 280° was probably a dicarboxylic acid (compare [8]).

The *p*-nitrobenzyl ester melted constantly at 142° to 142·5° after three crystallizations from alcohol and at 142° to 143° in admixture with the (KL) ester of the same m.p. (Found: C, 52·9; H, 3·1; N, 4·4; S, 20·6, 20·2. $C_{14}H_9O_4NS_2$ requires C, 52·7; H, 2·8; N, 4·4; S, 20·1 per cent.)

The methyl ester was obtained with diazo-methane and crystallized from aqueous alcohol and had m.p. and mixed m.p. 104° to 105° with the (KL) acid of the same m.p. (Found : C, 48·8; H, 2·6; S, 32·0. $C_8H_6O_2S_2$ requires C, 48·5; H, 3·0; S, 32·3 per cent.)

Final Purification of Liquid Thiophthen prepared from (a) *Sulphur and Acetylene*, (b) *Citric Acid.*

(*a*) Liquid thiophthen (10 g, freed from thionaphthen) of purity equivalent to specimen S_4 of Challenger and Harrison as used in the preceding experiments and prepared as described on p. 926 was added to styphnic acid (17 g) in hot methyl alcohol (60 cc). The styphnate separated on cooling and was crystallized successively from 35, 25, and 15 ml of methanol, when it melted at 130° to 131°. The thiophthen (S_5; 2 g) obtained on decomposition with sodium carbonate gave no trace of blue or green colour on polymerization with hydrogen bromide in glacial acetic acid, see p. 923. Further quantities, up to 3 g, of an equally pure product were obtained by further fractionation of the mother liquors.

(*b*) Liquid thiophthen (C) prepared from citric acid as described by Challenger and Harrison [1] and purified through the picrate was shaken for 15 min with saturated aqueous mercuric chloride. No blackening occurred and only a small amount of a white solid separated. The oil was then decanted, washed with water, and distilled. The m.p. of the picrate was then 137·5° and that of the styphnate 131°, in complete agreement with those of the corresponding compounds prepared from S_5 thiophthen. For mixed m.p. and further evidence of identity obtained with other derivatives, the table should be consulted. The precipitate obtained with mercuric chloride was insufficient for examination, and the nature of the trace of impurity thus removed is unknown. The white mercurated product did not melt or blacken at 300°.

The use of pure liquid thiophthen from the Sulphur Acetylene Reaction. Conversion to the Carboxylic Acid (KL) through the Methyl Ketone. The purest liquid thiophthen (S_5) was converted to the methyl ketone as before. After distillation in steam this melted at 115° to 116·5°, and after two crystallizations from alcohol constantly at 117°. (Found : C, 52·5; H, 3·1; Calc. for $C_8H_6OS_2$: C, 52·7; H, 3·3 per cent.) Oxidation with sodium hypoiodite as usual gave the carboxylic acid which, after five crystallizations from chloroform–acetone, formed fine white needles of constant m.p. 245° (slight decomp.).

The methyl ester after two crystallizations from methyl alcohol melted constantly at 106·5°. (Found : C, 48·9; H, 3·0; S, 32·4. $C_8H_6O_2S_2$ requires C, 48·5; H, 3·0; S, 32·3 per cent.)

The use of pure liquid thiophthen from the Sulphur–Acetylene Reaction. Conversion to the Carboxylic Acid (GL) by means of the Grignard Reagent. This preparation (see p. 926) was repeated using 4 g of the purest liquid thiophthen (S_5). This gave 1·4 g of an impure acid, m.p. 240° (decomp). Repeated extraction of this with chloroform left 0·2 g of the dicarboxylic acid and yielded 0·6 g of an acid of m.p. 243° (decomp). This was purified, with some loss, by boiling in 10 cc acetone for 3 min with 0·3 g of active charcoal. The *monocarboxylic acid* (0·15 g) was thus obtained as an almost

colourless solid of constant m.p. 245° (slight decomp). (Found: C, 46·1; H, 2·5. $C_7H_4O_2S_2$ requires C, 45·7; H, 2·2 per cent.)

The *methyl ester*, prepared as before using diazo-methane was purified by boiling with active charcoal in methyl alcohol, and melted constantly at 106·5°. (Found: C, 48·6; H, 3·1; S, 32·0. $C_8H_6O_2S_2$ requires C, 48·5; H, 3·0; S, 32·3 per cent.) These products were identical in m.p. and mixed m.p. with the corresponding derivatives prepared through the methyl ketone.

The Monocarboxylic Acid of Liquid Thiophthen (C). This thiophthen was prepared from citric acid and phosphorus trisulphide by the method of Challenger and Harrison [1] and purified through the picrate of m.p. 136° to 137° which was then decomposed with sodium carbonate. The resulting thiophthen (C) was converted to the methyl ketone, m.p. 115° to 116°, as recorded by these authors. Oxidation of this with iodine and sodium hydroxide as before gave thiophthencarboxylic acid KC, m.p. 245° (decomp), on crystallization from chloroform. Two more crystallizations from this solvent and one from water did not alter this m.p. It did not depress the m.p. (244° decomp) of thiophthencarboxylic acid (KL), see pp. 926 and 927. (Found: C, 45·5; H, 2·15; S, 34·5. $C_7H_4O_2S_2$ requires C, 45·7; H, 2·2; S, 34·8 per cent.) The *methyl ester*, prepared with diazo-methane formed colourless needles, m.p. 105°, alone and in admixture with the methyl ester of the KL acid. (Found: C, 48·9; H, 3·15. $C_8H_6O_2S_2$ requires C, 48·5; H, 3·0 per cent.)

On repetition of these two preparations using thiophthen (C_1) purified by shaking with aqueous mercuric chloride (see p. 923), the methyl ketone had m.p. 117°, the acid (KC_1), m.p. 245° (slight decomp), and the methyl ester, m.p. 106·5°. (Found for the methyl ketone: C, 52·5; H, 3·3; S, 34·8. $C_8H_6OS_2$ requires: C, 52·7; H, 3·3; S, 35·2 per cent. Found for the carboxylic acid: C, 45·5; H, 2·4. $C_7H_4O_2S_2$ requires C, 45·7; H, 2·2 per cent.)

Preparation of Thiophthen carboxylic acid (GC_1) from Citric Acid Thiophthen (C_1) and Magnesium Ethyl Bromide. The reaction was carried out as described for solid thiophthen by one of us and G. M. Gibson and for liquid thiophthen (S_5) in this communication. The acid on two crystallizations from acetone–chloroform melted constantly at 243° (decomp) raised to 244° on heating the aqueous solution of its potassium salt with activated charcoal. In admixture with the acid (KC_1) of m.p. 245° (slight decomp) the m.p. was 244° (decomp). (Found: C, 45·5; H, 2·2. $C_7H_4O_2S_2$ requires C, 45·7; H, 2·2 per cent.)

The Mono-iodo- Derivative of Pure Liquid Thiophthen (S_5). Liquid thiophthen (S_5; 3 g) in benzene (5 cc) was treated alternately in small portions during 15 to 20 min with iodine (5·6 g) and yellow mercuric oxide (4 g). The mixture was vigorously shaken and cooled in ice, the resulting solid separated, washed with benzene, dried with ether, and the washings shaken with aqueous sodium thiosulphate and then distilled in steam. The monoiodothiophthen solidified in the condenser and was crystallized to constant m.p. (55·5°) from 40° to 60° light petroleum and from methyl alcohol. It forms long needles having an odour resembling that of iodobenzene. Yield 2·8 to 3·2 g; 55 to 60 per cent.

Formation of Magnesium iodothiophthen and its reaction with Acetonitrile. Preparation of Methyl thiophthienyl ketone. Iodothiophthen (2 g) in dry

ether (8 cc) was added drop by drop during 10 min, in an atmosphere of nitrogen, to magnesium (0·2 g) covered with dry ether (2 cc), after addition of a trace of iodine and one drop of methyl iodide. A slow but steady reaction occurred, and two layers formed. The mixture was left for 5 hr when some magnesium still remained. Heating for 10 min caused no change. Acetonitrile (1 g) was then added and the mixture vigorously shaken. A white solid separated. The mixture was then acidified, the ether removed, and the residue distilled in steam. The volatile oil contained ketone, unchanged iodothiophthen, and some thiophthen (characterized as picrate). The ketone was not separated from the oil, but the aqueous distillate, when heated for 20 min with a saturated solution of 2 : 4-dinitrophenylhydrazine in 2N-hydrochloric acid, gave 0·05 g of a pure 2 : 4-dinitrophenylhydrazone, m.p. 265° to 266°. In admixture with the dinitrophenylhydrazone of the ketone prepared from liquid thiophthen (S_5) with acetyl and stannic chlorides (methylthiophthienyl ketone) of m.p. 267° to 268° the m.p. was 267°.

Acknowledgments.

The authors thank the Trustees of the Brotherton Scholarship and the Ministry of Education for maintenance grants to P. H. C. and R. E.

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ON THE INFLUENCE OF GLASS ON THE OXIDATION OF LOW VISCOSITY MINERAL OILS.

By D. J. W. KREULEN (Fellow) and F. G. KREULEN-VAN SELMS.

SUMMARY.

The influence of glass on the oxidation of mineral oils of low viscosity has been investigated. The presence of glass powder strongly affects the oxidation velocity of ordinary oils, but is unimportant in the case of white oils. Moreover, during the oxidation of white oils peroxides are formed, but with ordinary oils peroxide formation is negligible.

Investigations are reported in which the influence of the area of glass surface added and of temperature on the oxidation in the presence of glass is described.

The increase in viscosity of the oil during oxidation is discussed. A method for the determination of the carbonyl value of dark oils is given.

IN this paper previous work [1] on the oxidation of white oils, paraffins, xylene, and mixtures of xylene and white oils is continued, and technical oils which, unlike white oils, contain compounds with aromatic rings are investigated.

The difficulty met in oxidation experiments with technical oils is that they react so slowly with oxygen that there is always doubt whether the induction period is ended or not. A distinct increase of the typical oxidation constants is not generally observed, the only phenomenon from which it may be concluded that the oil reacts being the pronounced deepening of its colour.

In this paper experiments carried out with low-viscosity transformer and lubricating oils will be discussed. The oxidation of ordinary lubricating oils is still under study.

All experiments were carried out with oxygen in our standard apparatus [2] at a stirring velocity of 1400 r.p.m.

Our preliminary experiments, although they occupied some years, added practically nothing to our knowledge. They only provided several series of figures so frequently found in literature on this subject. It should be mentioned, however, that during certain experiments we noted the abnormal temperature susceptibility of the oxidation reaction (see also experiments D, H, K, and N). An oil which showed, after oxidation for 120 hr (140° C), an acid value of 0·17 mg KOH/g, an ester value * of 0·65 mg KOH/g, and a carbonyl value of 1·1 mg KOH/g had the same figures after oxidation for 6 hr at 150° C.

In order to ascertain whether chain reactions were involved, glass powder was added to an oil which was under reaction. After an oxidation period of 120 hr at 140° C the acid value of this oil was 0·17 mg KOH/g. Then 5 g of glass powder screened through a 30- and a 50-mesh-per-cm sieve was added, and a sharp increase in acid value resulted in the following oxidation period as is shown in Fig. 1. A similar increase was found for the ester value and for the carbonyl value.

* Ester value = saponification value minus acid value. In previous papers we wrongly mentioned "saponification number" when ester was meant.

Attention is directed to the fact that the increase in acid value due to the addition of glass powder did not proceed linearly, but lessened with time as seen in Fig. 1.

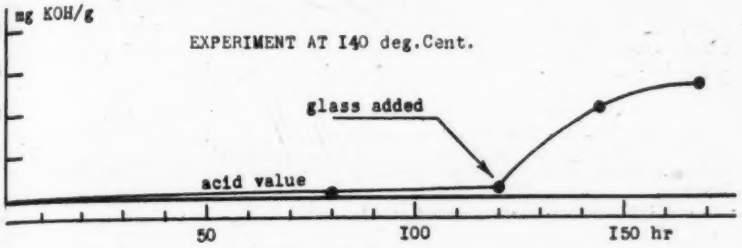

FIG. 1.

PRELIMINARY EXPERIMENT SHOWING THE INFLUENCE OF GLASS ON THE ACID VALUE OF A SLOW-OXIDIZING TRANSFORMER.

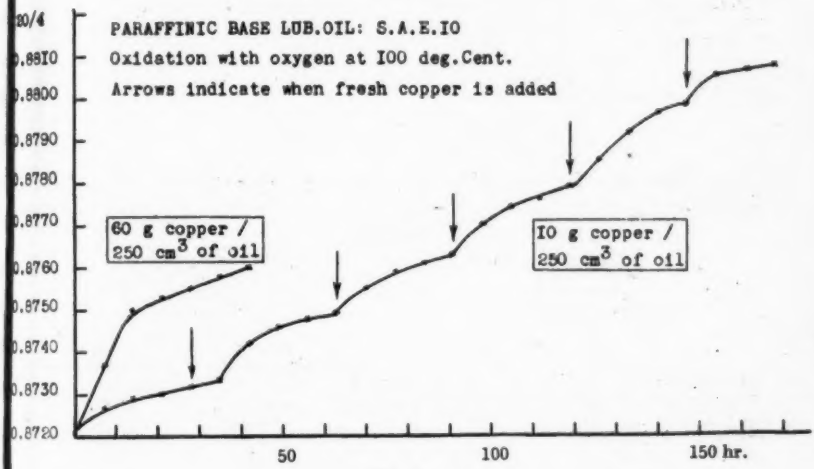
PARAFFINIC BASE LUB.OIL: S.A.E.IO
Oxidation with oxygen at I00 deg.Cent.
Arrows indicate when fresh copper is added
60 g copper / 250 cm3 of oil
I0 g copper / 250 cm3 of oil
50
100
150 hr.

FIG. 2.

INFLUENCE OF COPPER ON SPECIFIC GRAVITY OF OXIDIZING S.A.E.10 LUBRICATING OIL.

The phenomenon is apparently not restricted to glass powder, for in experiments with a low-viscosity lubricating oil (aromatic rings 9 per cent, naphthenic rings 16 per cent, paraffinic side chains 75 per cent) similar curves were obtained when copper filings were added and the specific gravity of the oil was plotted against reaction time. The results of one experiment are shown in Fig. 2.

After these observations it was decided to study this phenomenon more closely. Glass was used deliberately instead of copper in order to avoid the introduction of uncontrollable factors (copper salts) into the system.

In addition to the acid value and the ester value it seemed of interest to follow also the carbonyl value during the experiments. However, the methods which are described for the determination of the carbonyl value of organic compounds were not suitable as the oil acquired a dark colour during oxidation, and this made it impossible to titrate its solution. However, Dr S. H. Bertram provided us with the following method which, after some slight modifications, appeared to be quite suitable to our purpose :—

Method for the determination of the carbonyl value of dark oils.

Reagent. Dissolve 7 g of hydroxylamine hydrochloride in 150 cc of hot alcohol and add to it 100 cc of alcohol in which 5·5 g of KOH is dissolved. A precipitate is formed. Reflux for a moment and make up to 500 cc with alcohol and to 1 litre with benzene.

Filter : solution of hydroxylamine hydrochloride 0·1N.

Method. Weigh p g of oil ($p = 10$ g or less in order that at least 200 per cent surplus of hydroxylamine is present) into an Erlenmeyer flask. Add 50 cc of the reagent and reflux the contents of the flask gently for ½ hr. Transfer to a separating funnel and rinse the Erlenmeyer flask with 50 cc of water which is also put into the separating funnel.

The solution separates in two phases, the lower phase being the water phase. In order to improve separation some salt may be added. The lower phase is run into a titration flask, and the contents of the separation funnel are washed with 20 cc of water which, after separation, is added to the contents of the titration flask. Add 5 drops of methyl orange and 5 drops of methylene blue (45 mg per 50 cc) and titrate the solution with 0·1N-HCl.

If a cc of 0·1N-HCl are used and if in the blank test b cc of 0·1N-HCl are consumed the carbonyl value of the oils is :—

$$\frac{(b-a) \times 0{\cdot}1 \times 56}{p} \text{ mg. KOH/g.}$$

The constants for the oil used in the experiments are reported in Table I.

TABLE I.

Acid value	0·1 mg KOH/g
Ester value	0·2 mg KOH/g
Carbonyl value	0·1 mg KOH/g
Peroxides	Not present

	Before hydrogenation.	After hydrogenation.
Molecular weight	330	330
Aniline point, ° C	94·4	96·4
Sp. gr. 20/4	0·8644	0·8621
Refractive index D/20	1·4730	1·4714
Specific refraction	0·3246	0·3245
Viscosity (cs at 20° C)	33	—
Ring analysis :		
Aromatic rings, per cent	2	—
Naphthenic rings, per cent	35	37
Paraffinic side chains, per cent	63	63
Rings per molecule	2·4	2·4

The glass powder used was prepared by carefully sieving it through 65 and 200 Tayler sieves, then treating with bichromate–sulphuric acid for some days, washed till free from acid, dried, and resieved. From its average grain size (determined microscopically) and its specific gravity it was calculated that 560 mg of glass powder corresponded with 100 sq. cm. of glass surface.

From Fig. 1 it had been concluded that the activity of the glass decreases with time. Apparently the glass surface becomes contaminated and then loses its activity. This view is supported by the observation that the glass, after it has lost its activity, acquires a brownish colour. This colour cannot be removed by treating the glass with petroleum spirit or with benzene, but it can be removed by treating it with hot alcohol.

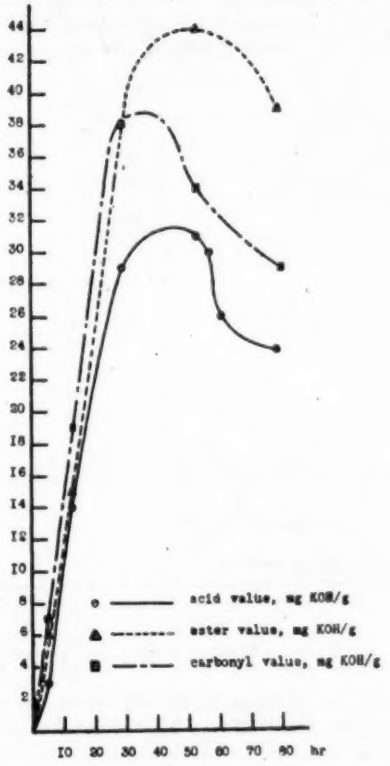

FIG. 3.

LONG-RUN OXIDATION EXPERIMENT (INDUCTION PERIOD EXCLUDED).

It would be ideal to conduct the experiments so that a continuous stream of clean glass enters the reacting system while an equivalent quantity of contaminated glass is removed simultaneously. This can be accomplished by using a moving glass braid belt which is regenerated immediately it leaves the oil.

This would, however, be complicated and therefore it was preferred to change the glass several times during an experiment. If the curves of

the increase of analytical values with time were smooth, then it was considered the replacement of the glass had been sufficient.

The following experiments are reported :—

Experiment L. This experiment was carried out to provide a general idea of the course of the acid value, ester value, and carbonyl value of an oil during prolonged oxidation.

750 sq. cm. of glass surface was present per 100 cc of oil and was renewed nine times during the experiment. Reaction temperature was 140° C.

As is shown in Fig. 3 the analytical values passed through a maximum. It is noteworthy that the viscosity passed through a maximum, too. The

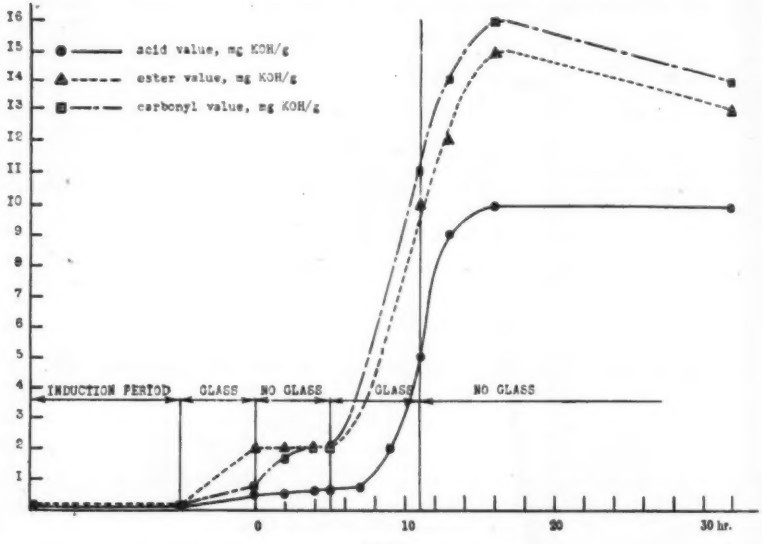

FIG. 4.

EXPERIMENT WITH ALTERNATING ADDITION AND REMOVAL OF GLASS.

fresh oil had a viscosity of 33 cs at 20° C; when the experiment ended after 79 hr the viscosity was 133 cs. However, after 57 hr oxidation the viscosity was 196 cs.

During the oxidation considerable amounts of water, carbon dioxide, and acetic acid were formed, but escaped when the reaction flask was opened to withdraw samples or to renew the glass. Therefore, as the degree of oxidation of the oil could reasonably be judged by its acid value, ester value, and carbonyl value, it was decided not to determine water, carbon dioxide, and acetic acid. However, it is assumed that they ran parallel with the other constants as the evolution of water, which is visible, practically stopped in the neighbourhood of the maximum of the curves.

The oxidized oil was stored in a glass flask. After some days a red-brown tenacious "resin" was deposited at the bottom of the flask. It dissolved again when the oil was heated.

Experiment C. Whether the reaction stops when the glass is removed and restarts when new glass is added was investigated in Experiment C. The results of this experiment, which was carried out at 140° C, are shown in Fig. 4.

When the test was made with glass (500 sq. cm. of glass surface per 100 cc of oil) the glass was renewed every 2 hr.

From Fig. 4 it appears that the reaction stops or starts according to the removal or addition of glass. Attention is directed to the fact that the

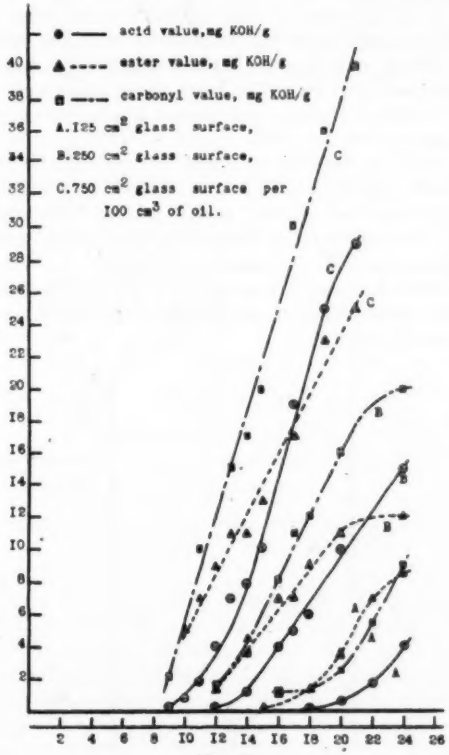

FIG. 5.

INFLUENCE OF AREA OF GLASS SURFACE ADDED ON REACTION.

reaction does not stop or start immediately the glass is removed or added. This was observed by us in several experiments, and it is possible that the delay observed after the addition of glass is intimately related to the lightening of the colour of the oil which, as was observed, goes with it.

Experiments B, D, and F. In these experiments the influence of the area of added glass surface was investigated. The tests were performed at 140° C with 125, 250, and 750 sq. cm. of glass surface per 100 cc of oil. The glass was renewed every 2 hrs.

From Fig. 5 it appears that the addition of more glass results in an increase in reaction velocity. This increase, however, is not in linear relation to the increase in glass surface, but is less, as is seen if the tangents of the angles of slope of the straight parts of the curves are compared.

An experiment was also carried out with 2250 sq. cm. of glass surface, and reaction velocities were found to be lower than when 750 sq. cm. of glass surface was added. Not much importance is attached to this experiment as the large amount of glass present clogged the stirrer through which the oxygen enters.

Experiments D, H, K, and N were carried out in order to investigate the influence of temperature on the reaction of oil in the presence of glass. In these tests 750 sq. cm. of glass surface was present per 100 cc of oil, and was renewed every 2 hr. The tests were performed at 120°, 130°, 140°, and 150° C with oil which had passed the induction period in consequence of a pre-treatment with oxygen at 140° C. As the length of the induction period is difficult to reproduce exactly, the zero values of these tests varied somewhat. This is, however, unimportant as the reaction velocity only is of interest and is judged from the slopes of the curves. In Fig. 6, therefore, the respective constants as a function of the reaction time (pre-treatment excluded) are plotted.

It is evident that the temperature susceptibility of the reaction velocity is extremely low, which suggests surface reactions which are governed by diffusion phenomena only.

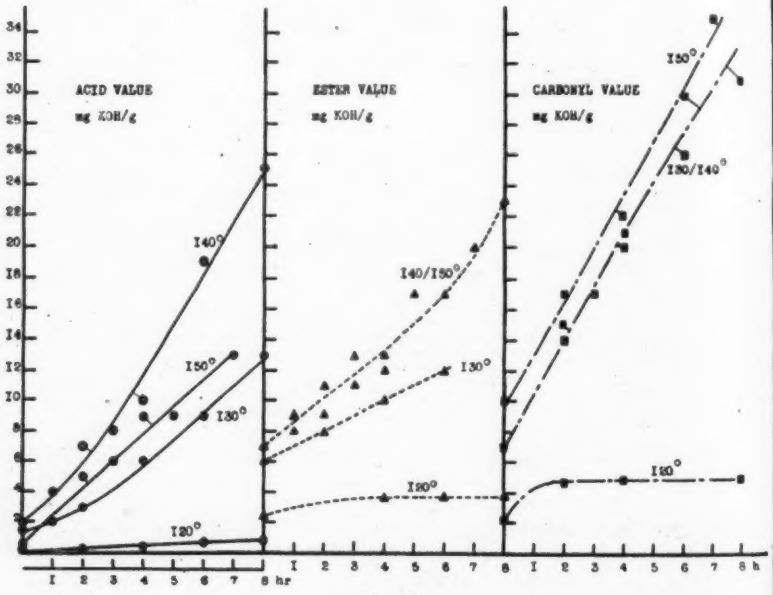

FIG. 6.

INFLUENCE OF TEMPERATURE ON OIL OXIDATION IN THE PRESENCE OF GLASS.

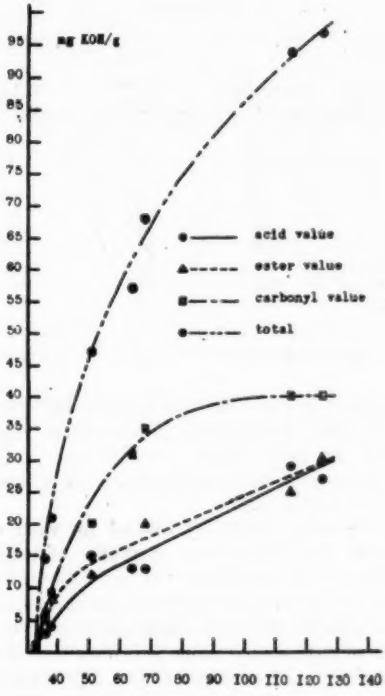

FIG. 7.

RELATIONSHIP BETWEEN ACID VALUE, ESTER VALUE, CARBONYL VALUE AND THE TOTAL OF THE THREE AND VISCOSITY.

It is noteworthy that the curves obtained at 120° C indicate practically no reaction. This suggests a minimum temperature which must be exceeded before appreciable reaction starts. It is possible that a similar phenomenon governs the reaction of oil without glass and that the presence of glass merely reduces the lower temperature limit at which appreciable oxidation starts.

It may be asked why the 150° curve of the acid value is beneath that of the 140° curve. This cannot be explained with certainty, but it may be that the formation of anhydrides has a part in it.

During this work the sharp increase of the viscosity as a function of the oxidation time was noted. Apparently this increase in viscosity is intimately connected with the formation of polar oxidation products.

In the description of Experiment L the parallelism of acid value, ester value, carbonyl value, and viscosity, the four of which passed almost simultaneously through a maximum, was mentioned. In Table II and Fig. 7 are given the oxidation constants and the viscosity of more or less oxidized oil.

TABLE II.

Viscosity 20° C, cs.	Acid value, mg KOH/g.	Ester value, mg KOH/g.	Carbonyl value, mg KOH/g.	Total, mg KOH/g.	Temperature at which the oxidation was performed, ° C.
33	0·1	0·2	0·1	0·4	Fresh oil
36	3	6	5·5	14·5	140
38	4	8	9	21	140
51	15	12	20	47	140
64	13	13	31	57	130
68	13	20	35	68	150
115	29	25	40	94	140
125	27	30	40	97	140

The figures are represented graphically in Figure 7.

It appears that there is a certain relationship between degree of oxidation and viscosity. This relationship is somewhat vague in the case of the acid value and the ester value. In the case of the carbonyl value and in that of the total of acid value, ester value, and carbonyl value, however, it is beyond any doubt.

Experiment Q. Previous work by one of us with white oils proved that when white oils (intramolecular mixtures of naphthenic rings and paraffinic side chains) are oxidized, the reaction proceeds appreciably without the presence of a catalyst, provided the induction period is passed.

As reported in this paper this is in sharp contrast to technical oils (intramolecular mixtures of naphthenic rings, paraffinic side chains, *and* aromatic rings). In our case even the presence of but 2 per cent of aromatic rings, which corresponds to less than one aromatic ring per molecule, appears to be sufficient to change considerably the activity of oil towards oxygen.

Yet there remained some doubt whether the previous experiments with white oil were quite comparable to the present ones, for the white oil was oxidized at 100° C while the present experiments were carried out at much higher temperatures.

A quantity of white oil (naphthenic rings 35 per cent, paraffinic side chains 65 per cent, and rings per molecule 2, 6) was, therefore, preoxidized at 100° C in the manner mentioned in previous papers in order to pass through the induction period. It was then divided into two equal parts, one of which was oxidized at 140° C without glass, while the other part was oxidized at the same temperature in the presence of 750 sq. cm. of glass surface per 100 cc of oil. This glass was renewed every 2 hr. The results are reported in Table III.

Although differences were found which are greater than the reproducibility of the test, it appears that the addition of glass does not affect essentially the reaction velocity of white oils.

Consequently, there is a distinct difference in behaviour between white oils and ordinary oils. Generally speaking it can be concluded that the presence of a boundary face (solid–liquid) strongly affects the oxidation velocity of ordinary oils, while the effect, if there is any, is relatively unimportant in the case of white oils.

TABLE III.

Reaction time, hr.	Without glass.			With glass.		
	Peroxides, mg O_2/g.	Acid value, mg KOH/g.	Ester value, mg KOH/g.	Peroxides, mg O_2/g.	Acid value, mg KOH/g.	Ester value, mg KOH/g.
0	0·2	0·1	2	0·2	0·1	2
2	3·1	2·5	8	2·9	1	9
4	5·7	9	17	6·6	8	19
6	5·1	17	25	5·3	17	29
8	3·7	31	34	5·1	27	45

Moreover, in the case of white oils peroxides are formed, while in the case of ordinary oils peroxide formation, if it occurs, remains quite negligible.

These differences must be related to the absence of aromatic rings in white oils and the presence of such rings in ordinary oils.

GENERAL OBSERVATIONS AND REMARKS.

It has already been mentioned that an induction period was observed during the oxidation of the oil. The length of this induction period amounted to 10 hr if the oil was oxidized at 140° C, provided glass was present. If glass was added after the oil had been pretreated with oxygen for 10 hr at the same temperature, reaction did not start immediately. The experiment had to be continued for another 10 hr with glass before reaction started. Apparently the presence of glass is an essential factor in bringing the oil through its induction period.

The reproducibility of the tests is rather poor and is estimated at about 10 per cent. In particular, the ester value is difficult to reproduce, and this might be caused by a transition of acids into anhydrides.

Sometimes deviations were observed which were quite inexplicable. Therefore all the experiments have not been reported, but only those which can be considered reliable.

In our previous papers on the subject of oil oxidation the data has been analysed with the aid of the Kreulen-ter Horst method in order to investigate whether a substance was formed directly or via one or more intermediate stages. This could not be done in the present investigation as surface reactions were being dealt with. In such cases, even if the formation of a compound runs undoubtedly via intermediate stages, its formation is a linear function of time.

It may be asked what is the practical value of the facts reported in this paper. There is but a remote relationship between these experiments and ordinary oil practice, but there is some practical background to the results. In support of this contention may be cited catalytic-cracking processes in which also hydrocarbons containing aromatic rings react via boundary surfaces.

Further, these experiments may indicate that excessive cleaning of the lubricating system of an engine may result in stronger acidification

of the oil. In fact, such phenomena has been observed in practice. It is also without doubt that the observations are of interest in the manufacturing of oxidized compounds to serve as oiliness improvers.

Finally the observations are of interest with respect to laboratory tests of oil stability, since glass is preferable to metals as it avoids the introduction into the oil of unknown chemical compounds.

ACKNOWLEDGMENT.

The authors wish to acknowledge the valuable help of J. Bosman, who devoted much of his spare time to this investigation.

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